भारतीय मानक
Indian Standard

IS 1448 [P : 65] : 2018

पेट्रोलियम एवं इसके उत्पादों की परीक्षण पद्धतियाँ

[पी : 65]

लुब्रिकेटिंग तेल के लिए ऑक्सीकरण परीक्षण

(तीसरा पुनरीक्षण)

Methods of Test for Petroleum and its Products

[P : 65]

Oxidation Test for Lubricating Oils

(Third Revision)

ICS 75.160.20

© BIS 2018

भारतीय मानक ब्यूरो
BUREAU OF INDIAN STANDARDS
मानक भवन, 9 बहादुरशाह ज़फर मार्ग, नई दिल्ली – 110002
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI-110002
www.bis.gov.in www.standardsbis.in

November 2018 **Price Group 3**

Methods of Sampling and Test for Petroleum, its Products, Gaseous Fuels and Lubricants Sectional Committee, PCD 01

FOREWORD

This Indian Standard [P : 65] (Third Revision) was adopted by the Bureau of Indian Standards on the recommendation of the Methods of Sampling and Test for Petroleum, its Products, Gaseous Fuels and Lubricants Sectional Committee and approval of the Petroleum, Coal and Related Products Division Council.

The first publication of this standard was based on IP 48, with the revision of IP 48 this standard was also subsequently revised in 1977, 1986 and 2011. Considerable assistance has also been derived from the ASTM D 4898 - 90 ‘Standard Test Method for Insoluble Contamination of Hydraulic Fluids by Gravimetric Analysis, in this revision.

The method defined is based on gravimetric analysis. In this revision, detailed shortened version of testing is given in Annex A, instead as a note. Details of calculation of evaporation loss and carbon residue increase, and test report are included.

In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 ‘Rules for rounding off numerical values (*revised*)’.

Indian Standard

METHODS OF TEST FOR
PETROLEUM AND ITS PRODUCTS

[P : 65]

OXIDATION TEST FOR LUBRICATING OILS

(Third Revision)

1 SCOPE

This standard [P : 65] covers the test method for the determination of tendency of lubricating oil to deteriorate on oxidation under specified conditions. A measure of the deterioration is obtained by comparison of the viscosity and carbon residue before and after oxidation. The test is not suitable for additive-type oils (other than those containing ashless additives) or those which form solid products or lose more than 10 percent by evaporation during the test.

WARNING — The use of this standard may involve hazardous materials, operations and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2 REFERENCES

The following standards contain provisions which, through reference in this text, constitute provisions of this standard. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent additions of the standards indicated below.

IS No.	*Title*
1448	Methods of test for petroleum and its products
[P : 8] : 2012/ ISO 4262 : 1993	Determination of carbon residue — Ramsbottom method (*second revision*)
[P: 25/Sec 1] : 2018/ ISO 3104 : 1994	Transparent and opaque liquids, Section 1 Determination of kinematic viscosity and calculation of dynamic viscosity (*second revision*)

3 PRINCIPLE

The sample is subjected to relatively severe oxidation conditions by heating to 200°C and passing air through it at 15 l/h for two periods of 6 h. After standing for 15 h to 30 h, the kinematic viscosity and Ramsbottom carbon residue of the oxidized oil are determined and compared with those from corresponding texts on the unoxidised oil. An alternative test procedure for the shortened version with a 12 h continuous oxidation period is given in Annex A.

4 REAGENTS AND MATERIALS

4.1 Petroleum Spirit, 60/80

4.2 Sulfuric Acid

Concentrated, of general purpose reagent grade or a non-chromium containing strongly oxidizing acid cleaning solution.

WARNING — Concentrated sulfuric acid and non-chromium containing strongly-oxidizing cleaning solutions are highly corrosive and potentially hazardous in contact with organic materials. Wear a full face-shield and full length protective clothing including suitable gloves. Avoid breathing vapor.

5 APPARATUS

5.1 Oxidation Tubes

Manufactured from borosilicate or neutral glass and conforming to the dimensions shown in Fig. 1.

5.2 Absorption Tubes

If required for reduction of objectionable atmospheric odors.

NOTE — The oxidation tube used in this method vents to the atmosphere and results in an unpleasant smell in some laboratories. This can be avoided by passing the issuing vapours through a tube containing water.

Absorption tubes shall conform to **5.1** and contain approximately 25 ml of water. The absorption tubes shall be mounted outside the bath and connected to the oxidation tubes. The length between the axis of the two tubes shall be 150 mm ± 50 mm. The connection between the oxidation and absorption tubes shall be as short as possible and jointed to the vessels by means of short flexible sleeves. To avoid evaporation of water,

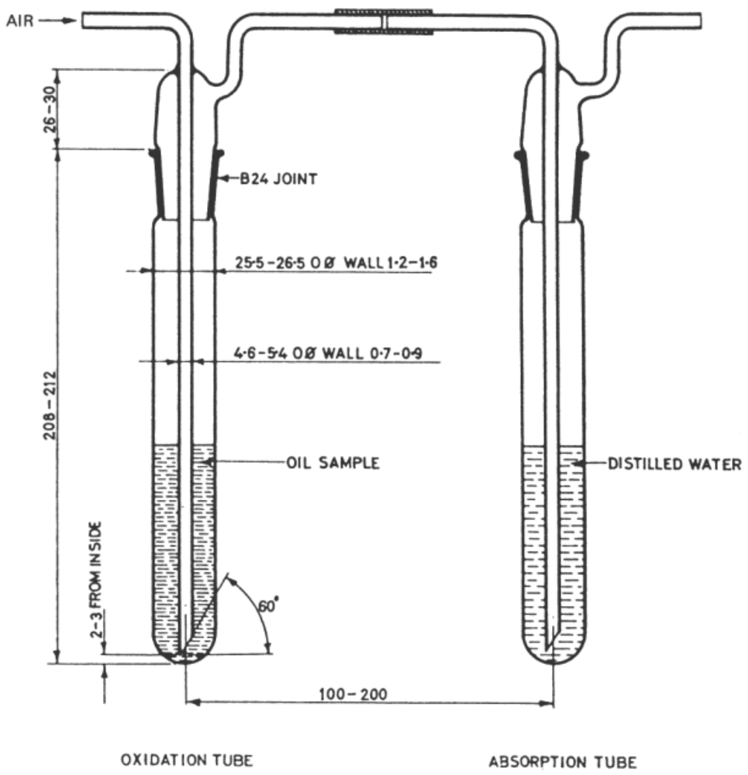

FIG. 1 OXIDATION AND ABSORPTION TUBES

ensure the absorption tube is protected from the heating bath by insulation.

NOTE — Silicone rubber sleeving has been found to be suitable for this purpose

5.3 Heating Bath

An aluminium alloy block heater or oil bath thermostatically controlled to maintain the oil in the required number of oxidation tubes at a temperature of 200°C ± 0.5°C (*see* Fig. 2). This temperature shall be read on a thermometer or a suitable temperature-measuring device (**5.5**), inserted to within 5 mm from the bottom of an oxidation tube containing 40 ml of oil (without an air supply) and placed in the heating bath. When using an aluminium block heater, the tubes shall be inserted into the holes to an overall depth of 150 mm. The depth of the holes in the heating part of the block shall be at least 125 mm and short metal collars, passing through the insulating cover and surrounding each oxidation tube, ensure heating over the 150 mm length of the tube. In the case of oil baths, the oxidation tubes shall be immersed to a depth of 137 mm in the oil and to an overall depth of 150 mm in the bath. For both types of heating bath the height of the oxidation tubes above the upper surface shall be 60 mm and the diameter of the holes shall be just sufficient to allow insertion of the specified tube. In case of slackness, a 25 mm diameter o-ring may be placed around the tube and pressed against the heater surface.

WARNING — The heating bath should be provided with a safety cut-out to prevent bath overheating.

5.4 Air Supply

From a suitable source of dry air, passed through a cotton wool or glass wool filter into a pressure stabilizer and then to the required number of oxidation tubes through any system capable of delivering 151/h ± 0.251/h to each oxidation tube, the rate being measured by a suitably calibrated flow meter. The flow rate shall be checked periodically by the use of a soap bubble flow meter, or an equivalent electronic device, connected to the exit of the oxidation tube or absorption tube if being used.

5.5 Thermometer

Shall comply with the following requirements or a suitable temperature-measuring device readable to ± 0.1°C and calibrated to better than ±0.1 °C.

Range	195 to 205°C
Graduation at each	0.1°C
Immersion	100 mm
Overall length	300 ± 10 mm
Stem diameter	5.0 to 8.0 mm
Bulb shape	Cylindrical
Bulb length	25 to 35 mm
Bulb diameter	Not greater than stem

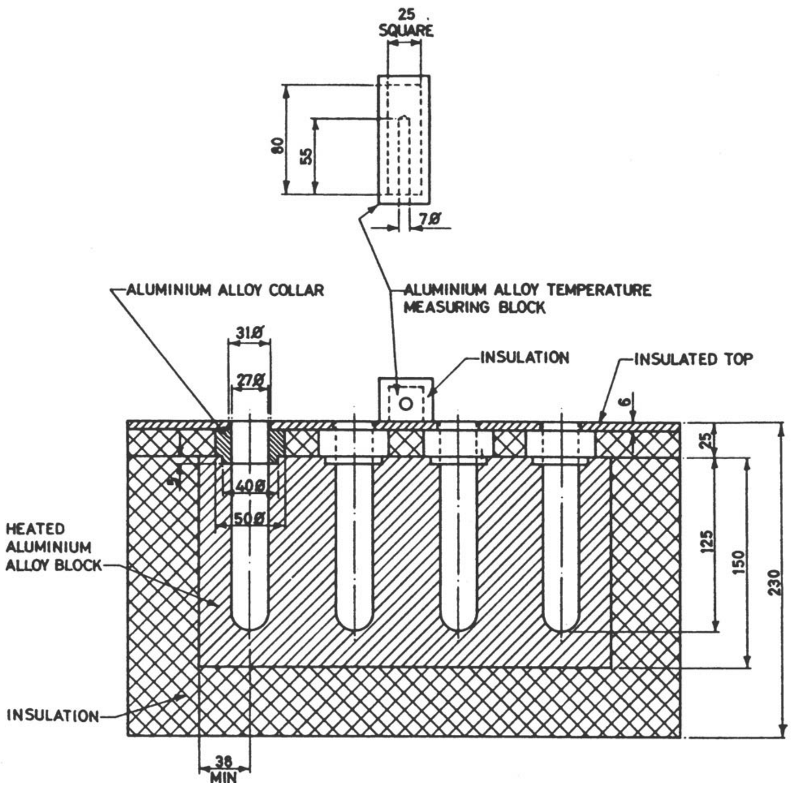

FIG. 2 TYPICAL METAL HEATING BATH

Length of graduated portion	70 to 110mm
Distance, bottom of bulb to 195°C graduation mark, *Min*	165 mm
Longer lires at each	0.5°C
Figured at each	1°C
Expansion chamber	Required
Top finish	Ring
Scale error not to exceed	± 0.2°C

5.6 Soap Bubble Flow Meter or an Equivalent Electronic Device

Capable of measuring the required flow rate. An example of a suitable soap bubble flow meter is shown in Fig. 3.

6 PREPARATION OF APPARATUS

The oxidation and absorption tubes shall be chemically cleaned.

NOTE — A satisfactory method is to wash out with petroleum spirit (**4.1**) and after drying, soak with concentrated sulfuric acid for a minimum of 16 h. If the concentrated sulfuric acid does not remove all deposits, repeat the acid cleaning with a non-chromium containing strongly-oxidizing acid cleaning solution (**4.3**). Drain and completely remove sulfuric acid by washing, first with tap water, then with distilled water. Dry the tubes in an air oven at 105 °C to 110 °C for at least 3 h and then allow to cool to room temperature in a desiccator in which they are kept until use.

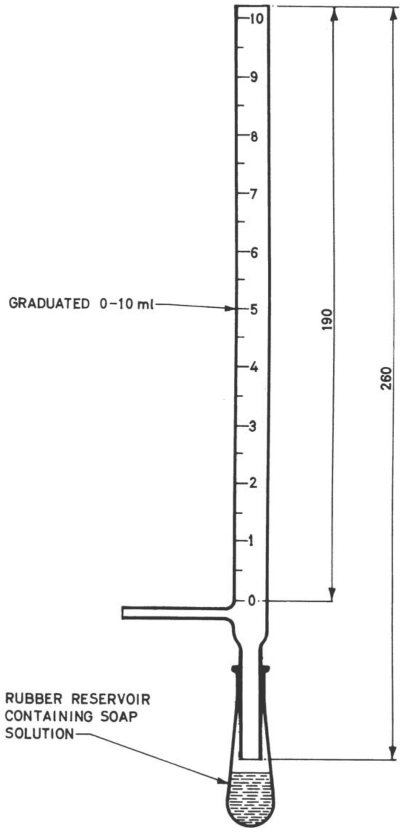

FIG. 3 SOAP BUBBLE FLOW METER

7 PROCEDURE

7.1 Weigh the oxidation tube and the ground-glass head to the nearest 0.1 g. Transfer 40 ml of the sample at room temperature into the oxidation tube, insert the ground-glass head and weigh to the nearest 0.1 g. Place the oxidation tube, ground-glass head and the sample in the heater at 200°C ± 0.5°C (*see* Note). If the absorption tube is being used, add 25 ml of distilled water and connect to the oxidation tube. Connect the air supply and adjust the air flow to 15 ± 0.251/h. To avoid evaporation of the water, the absorption tube should be protected from the bath by insulation.

NOTE — If a number of tubes are to be placed simultaneously in the bath, the tubes may be preheated for 5 min to 10 min at 180°C to 200°C in order to reduce the cooling effect of the oxidation bath.

7.2 When the oxidation bath has again reached 200°C ± 0.5°C, and not less than 10 min after introducing the oxidation tube, adjust the air flow to 151/h ± 0.251/h. Maintain this flow for the duration of the test. After 6 h ± 10 min, remove the oxidation tube from the bath.

7.3 Allow it to stand at ambient temperature for12 h to 18 h. Repeat the heating procedure for a further 6 h ± 10 min. At the end of this time, remove the oxidation tube from the bath and allow to cool to ambient temperature.

7.4 Weigh the oxidation tube, ground-glass and the oxidized sample to the nearest 0.1 g. Calculate the evaporation loss as described in **8.1**. The test is not suitable for evaluating a sample which loses more than 10 percent by evaporation during the test. Between 15 h and 30 h after completion of the oxidation periods, heat the oxidation tube in a boiling water bath and mix thoroughly to ensure the uniformity of the contents.

7.5 Determine, on the unoxidised oil, the kinematic viscosity at 40°C by IS 1448 [P : 25] and the Ramsbottom carbon residue by IS 1448 [P : 8].

7.6 Immediately after heating and mixing of the oxidized oil, charge a viscometer through a 75 pm filter as specified in IS 1448 [P : 25] and a Ramsbottom carbon residue coking bulb as specified in IS 1448 [P : 8]. Determine the kinematic viscosity at 40°C and the Ramsbottom carbon residue.

7.7 If successive determinations of the kinematic viscosity on the oxidized oil do not give consistent results within the determinability specified in IS 1448 [P : 25], report the oil as unsuitable for testing (**9**).

NOTE — The use of the same viscometer for the determination of the kinematic viscosity of the oxidized and unoxidised oil is recommended where practicable.

8 CALCULATION

8.1 Evaporation Loss

Calculate the percentage (m/m) evaporation loss, E, using the following equation:

$$E = (S - O) / (S - W) \times 100$$

Where,

W = is the mass of the oxidation tube and ground-glass head, in grams;

S = is the mass of the new sample, oxidation tube and the ground-glass head. in grams; and

O = is the mass of the oxidised sample, oxidation tube and ground-glass head, in grams

8.2 Viscosity Ratio

Calculate the viscosity ratio, VR, using the following equation:

$$V_R = V_2 / V_1$$

Where,

V_1 = is the kinematic viscosity of the unoxidised oil in millimetres square per second at 40 °C;

V_2 = is the kinematic viscosity of the oxidised oil in millimetres square per second at 40°C.

8.3 Carbon Residue Increase

Calculate the percentage (m/m) Ramsbottom carbon residue increase, CR,I, using the following equation:

$$C_{R,I} = C_{R,O} - C_{R,U}$$

Where,

$C_{R,O}$ = is the percentage (m/m) Ramsbottom carbon residue of the oxidised oil; and

$C_{R,U}$ = is the percentage (m/m) Ramsbottom carbon residue of the unoxidised oil.

9 EXPRESSION OF RESULTS

Report the following:

a) Kinematic viscosity of the unoxidised oil in square millimetres per second (mm^2/s) at 40 °C (*see* **7.5**);

b) Kinematic viscosity of the oxidized oil in square millimetres per second (mm^2/s) at 40°C (*see* **7.6**);

c) Viscosity ratio to the nearest 0.01 (*see* **8.2**);

d) Ramsbottom carbon residue of the unoxidised oil (*see* **7.5**);

e) Ramsbottom carbon residue of the oxidized oil (*see* **7.6**); and

f) Increase in carbon residue after oxidation (*see* **8.3**).

If consistent results are not obtained the oil shall be reported as being unsuitable for testing.

10 PRECISION

The following criteria should be used for judging the acceptability of results (95 percent probability) (*see* Note below **7.1**):

Viscosity ratio:

Level of result X	1.08 to 2.4
Repeatability	$0.2883\,(X - 1.0)$
Reproducibility	$0.5759\,(X - 1.0)$

Carbon residue increase:

Level of result X	0.2 to 2.4
Repeatability	$0.1258\,(X + 0.38)$
Reproducibility	$0.3392\,(X + 0.38)$

Where, X is the average of two results.

11 TEST REPORT

The test report shall contain at least the following information:

a) a reference to this standard;
b) the type and identification of the product tested;
c) the result of the test (*see* **9**);
d) any deviation, by agreement or otherwise, from the procedure specified; and
e) the date of the test.

ANNEX A

(*normative*)

An alternative test procedure for a continuous, 12 h oxidation period.

A-1 The full test period is three days and this may cause difficulties in refineries where production and laboratory facilities are continuous. In these circumstances, where the test is being used for production control, it is permissible to use a shortened version with the 12 h continuous oxidation period, followed by immediate determination of the viscosity and Ramsbottom carbon residue of the oxidized oil. However, it is necessary for the test to be carried out in full, in cases of dispute.

A-2 APPARATUS

Use the same apparatus as specified in **5**.

A-3 PROCEDURE

A-3.1 Follow the test procedure as given in **7.1**.

A-3.2 Follow the procedure as outlined in **7.2**, except use a continuous 12 h ± 10 min oxidation period, followed by immediate determination of the viscosity and Ramsbottom carbon residue of the oil (as specified in **7.4**, **7.5** and **7.6**).Omit procedure given in **7.3**.

A-4 CALCULATION

Determine the evaporation loss, viscosity ratio and Ramsbottom carbon residue in accordance with **8.1**, **8.2** and **8.3**.

A-5 PRECISION

Practical experience has shown that results obtained by the abbreviated version are normally within the repeatability and reproducibility of the full test procedure.

Bureau of Indian Standards

BIS is a statutory institution established under the *Bureau of Indian Standards Act*, 2016 to promote harmonious development of the activities of standardization, marking and quality certification of goods and attending to connected matters in the country.

Copyright

BIS has the copyright of all its publications. No part of these publications may be reproduced in any form without the prior permission in writing of BIS. This does not preclude the free use, in the course of implementing the standard, of necessary details, such as symbols and sizes, type or grade designations. Enquiries relating to copyright be addressed to the Director (Publications), BIS.

Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Catalogue' and 'Standards: Monthly Additions'.

This Indian Standard has been developed from Doc No.: PCD 01 (10329).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephones: 2323 0131, 2323 3375, 2323 9402 *Website*: www.bis.gov.in

Regional Offices:		*Telephones*
Central	: Manak Bhavan, 9 Bahadur Shah Zafar Marg NEW DELHI 110002	2323 7617 2323 3841
Eastern	: 1/14 C.I.T. Scheme VII M, V.I.P. Road, Kankurgachi KOLKATA 700054	2337 8499, 2337 8561 2337 8626, 2337 9120
Northern	: Plot No. 4-A, Sector 27-B, Madhya Marg CHANDIGARH 160019	265 0206 265 0290
Southern	: C.I.T. Campus, IV Cross Road, CHENNAI 600113	2254 1216, 2254 1442 2254 2519, 2254 2315
Western	: Manakalaya, E9 MIDC, Marol, Andheri (East) MUMBAI 400093	2832 9295, 2832 7858 2832 7891, 2832 7892

Branches : AHMEDABAD. BENGALURU. BHOPAL. BHUBANESHWAR. COIMBATORE. DEHRADUN. DURGAPUR. FARIDABAD. GHAZIABAD. GUWAHATI. HYDERABAD. JAIPUR. JAMMU. JAMSHEDPUR. KOCHI. LUCKNOW. NAGPUR. PARWANOO. PATNA. PUNE. RAIPUR. RAJKOT. VISAKHAPATNAM.

Published by BIS, New Delhi